EE143
Microfabrication Technology

Fabrication Process Manual

A detailed description of the EE143 NMOS microfabrication process for reference by students, TAs, and NanoLab staff.

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Updated by Daniel Teal, Fall 2023.

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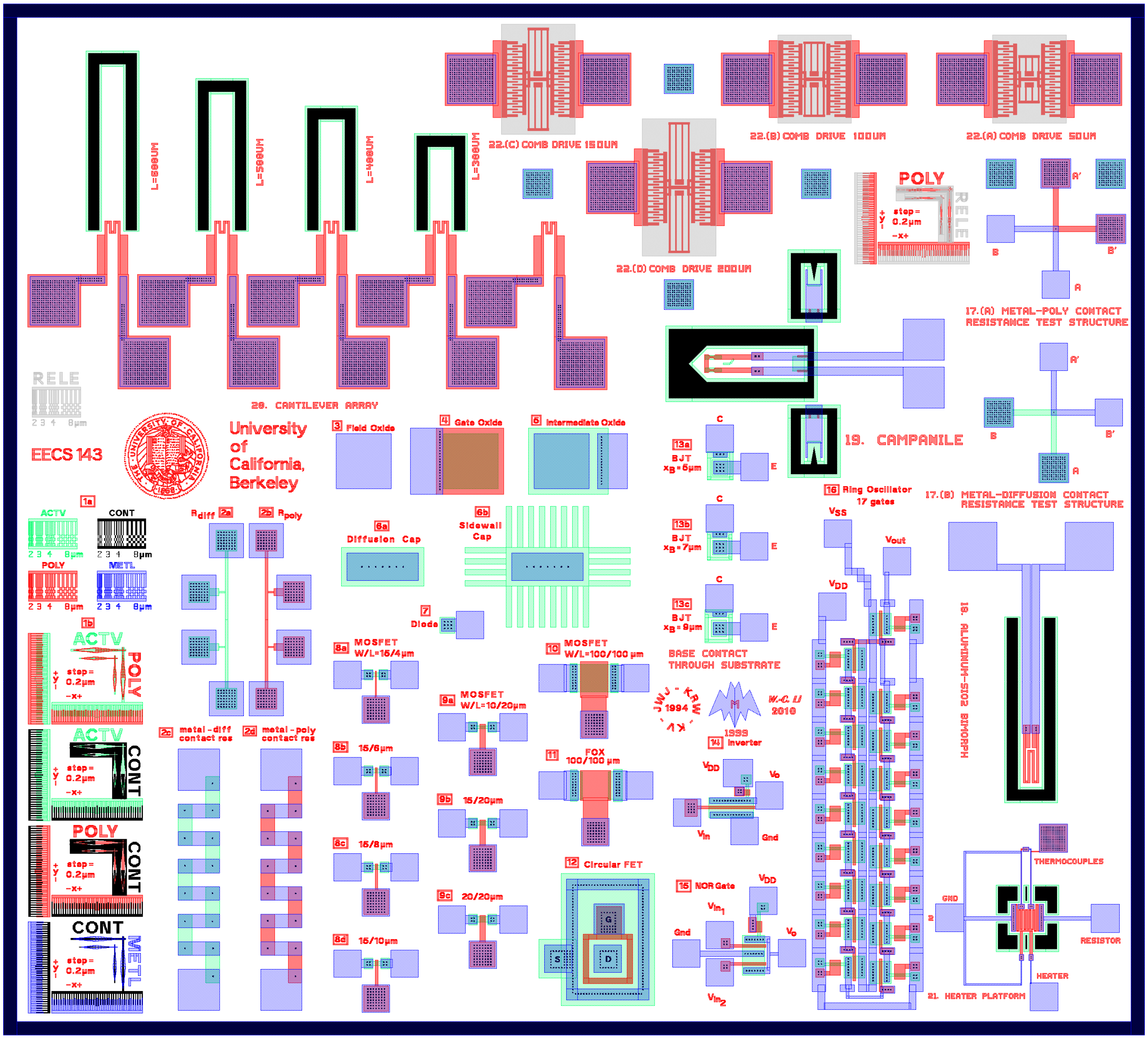
Chip Layout

The following figure depicts the layout of the EE143 chip. The ACTV, POLY, CONT, and METL photolithography patterns are colored green, red, black, and blue, respectively. The gray RELE layer is not used. The layout is 4.15 mm by 3.75 mm in size and tiled across the EE143 wafer such that each wafer has hundreds of copies. Its devices include:

- alignment marks and line/space measurement features (bottom left)
- MOSFETs of assorted sizes (bottom center)
- logic gates and a ring oscillator (bottom right)
- MEMS devices (top)

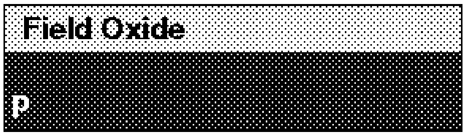
See the companion EE143 Characterization Manual for details of individual devices.



Process Flow Schematics

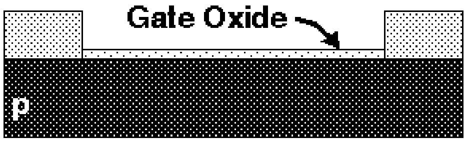
These figures are approximate cross-sections of a MOSFET on the wafer after each step. Note the class lab reports will require more accurate pictures (e.g., sidewalls are not perfectly vertical).

Step 1: Field Oxidation (done by TAs)

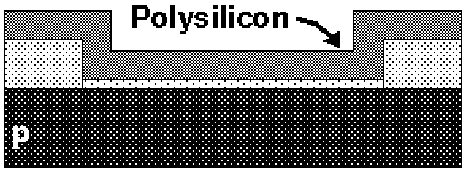


Step 2: Active Area (ACTV) Photolith. & Etch

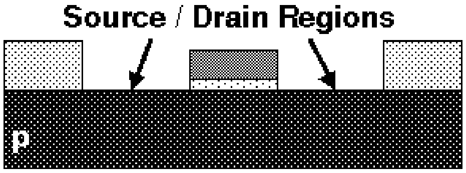
Step 3: Gate Oxidation



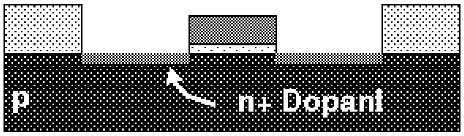
Step 4: Polysilicon Deposition (done by TAs)



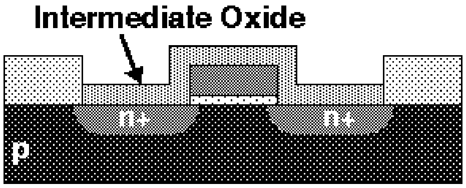
Step 5: Gate (POLY) Photolithography & Etch



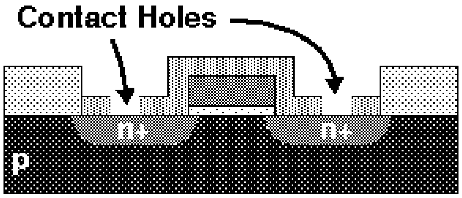
Step 6: Source/Drain Doping



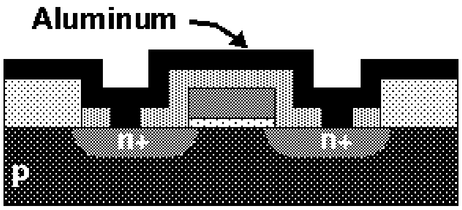
Step 7: Dopant Drive-in & Intermediate Oxidation



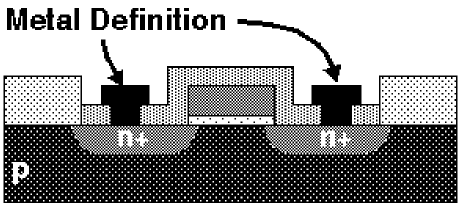
Step 8: Contact Hole (CONT) Photolith. & Etch



Step 9: Metallization



Step 10: Metal (METL) Photolithography & Etch



Detailed Process Flow

These are detailed instructions for each step of the fabrication process:

week 1	Lab Safety	
week 2	Step 1: Field Oxidation	(done by TAs/staff in NanoLab)
	Step 2: Active Area (ACTV) Photolithography & Etch	
week 3	Step 3: Gate Oxidation	
	Step 4: Polysilicon Deposition	(done by TAs/staff in NanoLab)
week 4	Step 5: Gate (POLY) Photolithography & Etch	
week 5	Step 6: Source/Drain Doping	
	Step 7: Dopant Drive-in & Intermediate Oxidation	
week 6	Step 8: Contact Hole (CONT) Photolithography & Etch	
week 7	Step 9: Metallization	
week 8	Step 10: Metal (METL) Photolithography & Etch	
wks 9–11	Device Characterization	

A typical semester schedule needs about 11 lab sessions (1 session/week) as indicated, with some steps performed by TAs (or NanoLab staff) between sessions. The TAs run an identical process in parallel at least one week ahead of the student labs both to test the process and to provide backups to replace broken student wafers.

Weeks 1–8 take place in the fabrication cleanroom. After this point, the wafers and devices on them are complete. Weeks 9–11 occur in the characterization lab.

See the companion EE143 Characterization Manual for device characterization details.

Step 1: Starting Materials & Field Oxidation (done by TAs)

MEASUREMENTS: furnace temperature & time, field oxide thickness.

1. Starting wafers
   (a) Begin with 3" p-type silicon wafers with $14\,\Omega\cdot$cm to $16\,\Omega\cdot$cm resistivity and $\langle 100\rangle$ crystal orientation. Each lab section should have one wafer per lab group (2-3 students each) and two control wafers (to monitor resistivity) for a total of ≈ 6 wafers per section. Also prepare $\approx 50\%$ more wafers to run during TA prelabs as backup.
   (b) Blanket implant $6.0\times10^{12}\,\text{cm}^2$, B11, $60\,\text{keV}$. The wafers and implantation are arranged by NanoLab staff.
   (c) Uniquely identify each of the wafers (including controls) by labeling them with a diamond scribe. Mark small numbers on the backside near the flat. Do not scribe off the edge of the wafer as this will cause the wafer to break. Scribing after field oxidation is acceptable.
2. Field oxidation (target $4500\,\text{Å}$ to $5000\,\text{Å}$) in NanoLab
   (a) Clean wafers (including controls) in NanoLab msink6 (standard piranha + HF). Refer to the msink6 manual on the NanoLab website (https://nanolab.berkeley.edu) for more detailed information. A brief summary follows:
      i. If the new wafers arrive in a vendor-sealed package, the standard pre-msink6 clean in msink8 can be skipped.
      ii. Transfer 3" wafers into the clean 3" Teflon cassettes kept in the drawer marked "EE143" next to msink6. Matching cassette handlers are in the same drawer.
      iii. Follow the standard msink6 procedure to clean the wafers in piranha, 25:1 HF, HCl, and HF again, with water rinses in the QDR station between each step.
      iv. Because there is no 3" automatic spin rinse dry (SRD) system in the NanoLab, use an N_2 gun to manually dry the wafers. Remember only Teflon tweezers or clean wands should hold the wafer post-clean / pre-furnace.
   (b) Oxidize wafers (including controls) at $1000\,^{\circ}$C for 5-90-5 minutes (dry-wet-dry).
      - Use a MOS clean furnace (e.g., tystar2 with recipe 2WETOXA). Do not use tystar3 or 4 which are not clean enough for this application.
      - Before oxidation, please ask NanoLab staff to help put the 3" quartz boat (stored in the dedicated EE143 drawer near msink6) into the furnace.
      - In 2021 we ran 2WETOXA at $1000\,^{\circ}$C with a $125\,$min wet oxidation step which grew $522\,$nm oxide. In 2023 we ran the same for $88\,$min and obtained $445\,$nm.
      - After oxidation, wait at least $10\,$min for the wafers to cool down sufficiently (to avoid melting plastic) then load them directly into a plastic 3" wafer box or tray. Unload with a vacuum wand and face shield as is normal for the furnaces.
   (c) Measure field oxide thickness on Nanospec.

Step 2: Active Area (ACTV) Photolithography & Etch

MEASUREMENTS: dehydration bake temperature & time, HMDS time, photoresist spin speed & time, soft bake temperature & time, exposure time, development time, etch time, pictures of ACTV line/space structure (a) in photoresist and (b) after PR strip, control wafer resistivity.
CONTROLS: Do not do lithography on control wafers, but HF etch to remove oxide and measure resistivity. This will confirm the starting resistivity of all wafers.

1. Apply photoresist.
    - (a) Dehydrate wafers in an oven for at least 10 min at $110\,^{\circ}$C to remove adsorbed water vapor to improve photoresist adhesion.
    - (b) Place wafers in HMDS vapor for at least 5 min to form an HMDS monolayer, which improves adhesion between silicon dioxide and photoresist.
    - (c) Spin photoresist.
        - i. Ensure the spinner is set to 3000 RPM spin speed for 30 s, which should result in a photoresist film about 1 µm thick.
        - ii. Obtain MiR 701 photoresist (typically stored in the refrigerator; let it warm to room temperature before using).
        - iii. Place a wafer onto the spinner chuck.
        - iv. Actuate the black toggle switch under the front of the spinner to turn on the chuck vacuum to hold the wafer in place.
        - v. Blow across the wafer with the air gun to remove any dust.
        - vi. Use a plastic pipette to dispense photoresist on the wafer. Covering a majority of the wafer surface often improves spinning uniformity as this spinner has no slow spin step to spread out the resist.
        - vii. Start a spin cycle by pressing the large button on the foot pedal (the small pedal button stops in the middle of the spin cycle if need be).
        - viii. When the spin cycle is complete, toggle the vacuum off and remove the wafer.
        - ix. Return the MiR701 photoresist to storage.
    - (d) Soft bake the photoresist by placing the wafer on a $90\,^{\circ}$C hotplate for 1.5 min. This improves adhesion, evaporates most of the PR solvent to prevent it sticking to the mask during lithography, and reduces removal of unexposed resist during development. Note the surface of a hotplate is often actually $5\,^{\circ}$C to $10\,^{\circ}$C cooler than the set temperature.
2. Expose the wafer using the ACTV mask. Note the pattern does not have to be precisely aligned to anything yet (alignment will be important for future lithography steps).
    - (a) Set the proper exposure time on the Kasper mask aligner. Typical exposure times are 3 s to 6 s depending on current lamp output power and wafer surface reflectivity. This is a sensitive parameter so TAs will find an optimal exposure time during the prelab. The time will also depend on the reflectivity of the wafer (e.g., in later steps, the polysilicon is less reflective than the field oxide and may need a higher exposure time).

Underexposing is much worse than overexposing because it may result in incomplete resist development. Historic values: $3\,\mathrm{s}$ with new lamp ($70\,\mathrm{mJ/cm^2}$) Spring 2019, $4.5\,\mathrm{s}$ to $5.8\,\mathrm{s}$ Fall 2022.

   (b) Load the wafer into the Kasper mask aligner.
   (c) Expose the wafers.

3. Develop the photoresist.
   (a) Post-exposure bake (PEB) the photoresist by placing the wafer on a $90\,^{\circ}\mathrm{C}$ hotplate for $1.5\,\mathrm{min}$. This increases the solubility of the resist in developer.
   (b) Develop the wafers by dipping them in a beaker of MF-26A developer and agitating gently. Development typically takes about $60\,\mathrm{s}$. Observe the reddish liquid forming near the wafer. It is the dissolved photoresist.
   (c) Rinse ($30\,\mathrm{s}$ to $60\,\mathrm{s}$) in 3 successive DI water rinse beakers with gentle agitation.
   (d) Dry the wafers with an air gun. It is very easy to break wafers during this step, so use caution. It may help to dry over a Technicloth at the sink.
4. Inspect the photoresist pattern under a microscope to ensure development is complete. Take a picture of the ACTV line/space structure for later computerized measurement of line and space widths.
   - **IMPORTANT:** The photoresist should have sharp clean lines and flat colors. Large splotches or color gradients indicate incomplete development, likely due to underexposure or missing PEB. This is a bad etch mask and should be stripped and reapplied.
5. Etch the field oxide (using the PR as a mask).
   (a) Pour buffered 5:1 HF (BHF) into a **plastic** beaker to the fill line (the TA will do this). Buffered HF is a mixture of NH_4F (ammonium fluoride) and HF 5:1. Its etch rate of thermal SiO_2 is $\approx 1000\,\mathrm{\AA\,min^{-1}}$ at $25\,^{\circ}\mathrm{C}$. BHF is used rather than plain diluted HF because the buffer keeps the strength and thus etch rate closer to constant.
   (b) Dip wafers in water for at least $10\,\mathrm{s}$ (rinse beaker 1 is acceptable) to wet the surface. Because of the surface tension of BHF, air bubbles can sometimes get trapped on the wafer surface if the surface is completely dry, leading to localized areas where the oxide is not removed.
   (c) Dip the wafer into the buffered 5:1 HF for a sufficient amount of time to etch the oxide, plus a 15% overetch. The overetch is performed for process latitude (i.e., because the oxide thickness and the etch rate both may vary across a wafer and among wafers). Etching is complete when the etchant "beads" on bare Si (SiO_2 is hydrophilic, but bare Si is hydrophobic).
      - Overetching in this step is possible and will result in larger-than-desired ACTV features. Fortunately, this does not affect device operation much so this step is not very sensitive.

   (d) Rinse in DI water 3 successive times in separate rinse beakers (agitating gently), then spray rinse.
   (e) Blow dry the wafers with air gun.

6. Inspect the etched area with the microscope and/or Nanospec to ensure etch is complete, i.e., nothing looks unusual and needs fixing. It is not necessary to save a picture here.

7. Strip the photoresist.
   (a) Dip wafer in a Remover 1165 bath for $5\,\mathrm{min}$ to dissolve the photoresist. Note the wafer can be left in this bath for any longer amount of time (even weeks!) because the Remover 1165 will not etch any of the other materials on the wafer.
   (b) Rinse the wafer in a beaker with IPA for 1 min to remove residual photoresist.
   (c) Rinse in DI water for 1 min in each of 3 dedicated rinse beakers successively.
   (d) Blow dry.

8. Inspect the now-etched oxide under a microscope. Take a picture of the ACTV line/space structure. Note its line and space widths may be different now than when measured in the PR pattern due to oxide overetch.

9. Use the same field oxide etch procedure to strip the oxide off the control wafers (which should now be blank silicon). Measure their resistivity using the 4 point probe. Record this measurement as the initial resistivity of the wafers.

Step 3: Gate Oxidation

MEASUREMENTS: furnace temperature & time, gate oxide thickness.
CONTROLS: Gate oxide should be grown on the control wafers to measure the oxide thickness. In TA prelab, also grow 100 nm oxide on 2–4 separate wafers as monitors for polysilicon deposition.

1. 2-4 days before first (pre)-lab session, TA's should ask NanoLab staff to turn on and TCA clean the furnace (see appendix, page 22)

2. Clean wafers (including controls) before loading into furnace.

   (a) Prepare piranha solution in a **Pyrex (glass)** beaker as follows: measure 10 parts H_2SO_4, and very slowly add 1 part H_2O_2. This mixture is self-heating. When cool it may be refreshed by slowly adding 1 part H_2O_2. Note it helps to use a large beaker so many wafers can be cleaned in this at the same time.

   (b) Prepare 10:1 BHF in a **plastic** beaker.

   (c) Immerse wafers in piranha solution for up to 10 minutes. Do not agitate the wafers as this is hard to do safely. Piranha removes organic contaminants by oxidizing (burning) them and metals by forming soluble complexes.

   (d) Rinse wafers in 3 successive DI water baths for 1 minute each.

   (e) Dip in 10:1 BHF for 20 seconds, agitating gently. This HF dip removes the native oxide.

   (f) Rinse wafers in 3 successive DI water baths for 1 minute each.

   (g) Using teflon tweezers, place all wafers into 3" white Teflon cassette then place into Vertex spin dryer until resistivity meter indicates $10\,\mathrm{M\Omega}$ to $15\,\mathrm{M\Omega}$ or for 10 minutes.

      - If the spin dryer is not working, dry the wafers by hand with an air gun, but wear clear poly gloves in addition to full chemical PPE in order to minimize contamination.

3. Oxidize wafers (and controls) $700\,\text{Å}$ to $900\,\text{Å}$ in furnace.

   (a) Load wafers into furnace. Use tube #3 (bottom) at $1100\,^\circ$C.

      i. Set the furnace gases as follows. Enable nitrogen and oxygen flow by turning the nitrogen knob to BUBBLER (this should already be set) and the oxygen knob to point up. Use the flowmeters to set $N_2 = 40$ sccm and $O_2 = 15$ sccm, which is 90% N_2 and 10% O_2.

      ii. At loading/cooling table, use vacuum wand to transfer wafers onto glass boat, then use short glass rod to carefully pull the boat into the cylindrical glass carrier.

         - Tip: Face the backside of the wafers towards the hole in the carrier through which the glass rod will transfer the boat to the furnace tube. Also put the wafers closer to the far side of the boat from the carrier hole. This way the glass rod is less likely to slip off the boat and hit a wafer, and if it does, it will hit the backside, not hurting your devices. Unless you break the wafers, which will hurt your devices.

   iii. Remove the end cap from furnace tube (holding it with a heat-insulating glove) and place it on the fire brick on the nearby table.
   iv. Attach the cylindrical carrier to the end of the tube and push the boat into the tube very slowly (1" every 10 seconds) to avoid thermal shock which could break the boat. Push the boat in to a depth predetermined by the TAs.
      - **CAREFUL:** Do not push the cylindrical carrier tightly onto the tube, because it can get stuck.
   v. Remove the cylindrical carrier and replace the end cap on the tube, ensuring its gas outlet is pointing left, toward the exhaust (scavenger) opening.
      - Do not push the end cap tightly onto the tube, because it can get stuck.

(b) Dry oxidize wafers. Dry (rather than wet) oxidation is done for a higher quality oxide with a more controlled thickness and a better oxide-silicon interface.
   i. Change the gas flows to $N_2 = 0$ sccm, $O_2 = 120$ sccm.
   ii. Let the wafers oxidize for approximately 60 min (time determined by TAs).
      - The EE143 furnace normally grows less dry oxide than would be expected by the Deal-Grove model. The target is $700\,\text{Å}$ to $900\,\text{Å}$.

(c) Anneal wafers. This allows the silicon atoms to diffuse and heal some of the damage done during oxidation.
   i. Turn the O_2 flowmeter to 0 sccm, and the O_2 knob to OFF. Set $N_2 = 40$ sccm.
   ii. Anneal in N_2 for 10 min.

(d) Unload wafers from furnace.
   i. Pull wafers out in the same 40 sccm N_2.
   ii. Remove the end cap, attach the cylindrical carrier to the end of the tube, and pull the boat out slowly (1" every 10 seconds) into the carrier. Place carrier with boat onto loading/cooling table, and replace furnace end cap.
   iii. Set the furnace gas to 120 sccm N_2. This keeps the tube clean in between runs. Note the N_2 is supplied building-wide so it is not likely to run out (unlike O_2 and forming gas, which are supplied by cylinders in the chase behind the furnace).
   iv. At the loading/cooling table, use short glass rod to push wafer boat out of cylinder carrier onto glass half-cylinder boat holder, and let cool for 5 min to 10 min.
   v. Remove the wafers very carefully! This step has broken many wafers!
      - The device wafers should be unloaded into a clean box to bring directly to the NanoLab for polysilicon deposition. The control wafers can be placed into the usual wafer box for the lab section.

4. Measure gate oxide thickness on control wafers with Nanospec.

Step 4: Polysilicon Deposition (done by TAs)

MEASUREMENTS: furnace temperature & time, polysilicon thickness & sheet resistance.
CONTROLS: The two control wafers for each lab section are not used, but the extra oxidized wafers prepared in Step 3 are used as monitor wafers for the polysilicon deposition.

1. Clean wafers in NanoLab msink6.
    - **CAUTION:** DO NOT use HF! It will etch the gate oxide, which we do not want to change the thickness of.
    - Use piranha and HCl only. Otherwise follow the same steps as in Step 1 on page 5.
    - If wafers were brought in a clean box directly from the gate oxidation furnace, an msink8 clean may be skipped.
2. Deposit $4000\,\text{Å}$ Phosphorus-doped polysilicon in tystar10 using the standard recipe.
    - Time was about 3 hours 30 minutes in tystar10 in Fall 2013.
    - The standard tystar10 process temperature is $615\,°$C.
    - 3" closed boats are available for this process.
    - Wafers should be loaded in the center of the boat for tystar10. Try to avoid the first and last two work position in the boat. Do not remove the 4" dummies and load the 3" wafers in the same slots. To load the 3" wafers efficiently, use the wand to suck the front side of the 3" wafers and then slide the 3" wafer into the slot with their backs against the 4" dummies. The 3" wafers stay nicely and they keep the vertical position during the deposition.
    - Try to include oxidized 3" monitor control wafers (prepared in TA prelab for Step 3) on either side of the 3" device wafers to measure the polysilicon thickness. These monitor wafers should have $1000\,\text{Å}$ SiO_2 on them.
    - The furnace uses silane to supply silicon and phosphine for phosphorus for n-type doping. Note the polysilicon will be further n-doped during steps 6 and 7.
3. Measure polysilicon thickness on a monitor control wafer via Nanospec (poly on oxide program).
4. Measure polysilicon sheet resistance via 4-point probe on a monitor control wafer.

Step 5: Gate (POLY) Photolithography & Etch

MEASUREMENTS: pictures of POLY line/space structure (a) in photoresist and (b) after PR strip, picture of POLY-ACTV alignment mark, polysilicon etch time, gate oxide etch time.
CONTROLS: Control wafers are not used during this step.

1. Apply photoresist to the wafer, expose it, and develop via the same process used in Step 2 on page 6. This time, expose the POLY mask and align to the ACTV layer on the wafer. Note it may take some time to learn how to align the layers properly.

2. Save microscope images of the POLY line/space and POLY-ACTV alignment patterns.

3. Hard bake the photoresist-coated wafer in a $110\,^{\circ}\mathrm{C}$ oven for $10\,\mathrm{min}$. Hard baking cross-polymerizes the PR polymer, making the photoresist physically hard, more adhesive, and less permeable to chemicals.

4. Etch polysilicon.

    (a) Prepare a **plastic** beaker of 10:1 BHF, and a **plastic** beaker of a premixed silicon etchant obtained from the NanoLab (composition: 64% HNO_3 / 33% H_2O / 3% NH_4F). This etches silicon at $\approx 2000\,\text{Å}\,\mathrm{min}^{-1}$ (it also etches SiO_2, at a slower rate).

    (b) Dip wafers in water (rinse tank 1 is acceptable) to wet the surface.

    (c) Dip wafers into the 10:1 BHF solution for $20\,\mathrm{s}$ to remove any native oxide, which is etched more slowly than polysilicon.

    (d) Rinse wafers in 3 successive water rinse beakers.

    (e) Immerse wafers in the silicon etchant. Etch time is often around $2.5\,\mathrm{min}$ for a $4000\,\text{Å}$ film. To determine when to stop the etch, watch the color of the polysilicon, especially visible on the wafer backside, which changes as it is etched. When the color has stopped changing completely the etch is done. Stop the etch immediately (or even a few seconds before this point) and proceed with the rinse.

        - **CAUTION:** overetching is extremely easy. This will make the MOSFET gates smaller, potentially to the point they do not work.

    (f) Rinse the wafers in 3 successive DI water rinse beakers and dry with air gun.

5. Inspect etching for completion under microscope. Pattern edges should be sharp and clear.

6. Clear the source/drain areas by HF etching the gate oxide, using the same photoresist mask as for the polysilicon (this simplifying trick is called a "self-aligned gate").

    (a) Wet wafers in DI water, then etch wafers in 10:1 BHF for $\approx 5\,\mathrm{min}$ to remove the gate oxide in the active areas. Note this will likely overetch the oxide a little. This is preferred because it is critically important all the oxide is removed so the dopant in Step 6 can contact the active area silicon directly. But don't overetch the field oxide!

    (b) Rinse the wafers in 3 successive DI water rinse beakers and dry with air gun.

7. Strip photoresist with Remover 1165 using the same process as in Step 2 on page 8.

8. Save microscope images of the POLY line/space pattern.

Step 6: Source/Drain Doping

MEASUREMENTS: furnace temperature & time, resistivity after spin-on glass strip.
CONTROLS: The control wafers are extremely important in this step. They should be doped at the same time as device wafers. Upon removing all wafers from the furnace, a single control wafer should have its spin-on glass stripped and resistivity checked to confirm it was doped. If not (very possible if native oxide wasn't removed), the device wafers and remaining control wafer can be put back into the furnace, potentially after re-applying spin-on glass if need be. The remaining control, which will be similar to the doped areas of device wafers, will be used to measure resistivity after drive-in.

1. Clean wafers with the same pre-furnace clean as Step 3 on page 9 (piranha & 10:1 HF).
    - **IMPORTANT:** Use HF dip to remove all native oxide. If any oxide is present, it will block dopants from diffusing from the spin-on glass into the silicon.
    - Don't overetch with HF or too much field oxide will be removed.
    - **IMPORTANT:** Minimize time between this clean, the following bake, and applying spin-on glass in order to minimize native oxide growth.
    - Now that the wafers are furnace-clean, only handle them with teflon or vacuum tweezers, not the standard metal tweezers.
2. Bake wafers in oven for $5\,\mathrm{min}$ at $200\,^{\circ}\mathrm{C}$ in order to completely remove any water on the surface, which might prevent the spin-on glass coating the wafer uniformly.
    - In oven, put the wafers on glass slides to prevent contamination. Be careful with Teflon, which flows at this temperature.
3. If completing Step 7 (drive-in and intermediate oxidation) in the same lab session, this is about the right time to turn on the bubbler for that step.
4. Use spinner to spin Filmtronics Phosphorosilica spin-on glass (SOG) at $3000\,\mathrm{RPM}$ for $20\,\mathrm{s}$.
    - This spin-on glass contains a high concentration of phosphorus that will diffuse into the wafer to dope it.
    - Less than half of a pipette-full of SOG is needed because its viscosity is so low.
    - Check the manufacturing date beforehand. SOG expires after six months due to increased viscosity.
5. Bake wafers at $200\,^{\circ}\mathrm{C}$ for $15\,\mathrm{min}$ in oven to remove solvent from spin-on glass.
6. Diffuse dopant from spin-on glass into wafers via furnace (at $1050\,^{\circ}\mathrm{C}$).
    (a) Load wafers into the glass boat.

   (b) Use furnace tube #2 (center) at $1050\,^{\circ}$C. Set N_2 to 40 sccm (and the top-left knob should be at "bubbler" like usual). Turn oxygen on (top center knob pointing up) with O_2 flowmeter at 15 sccm. This corresponds to 10% O_2 and 90% N_2, and is constant during push, deposition and pull. No water vapor should be used yet (bottom center knob should be at "furnace") (that will be used for Step 7, drive-in and intermediate oxidation).
   (c) Push in wafers at 1 inch every 10 seconds.
   (d) Hold 10 minutes in hot zone.
   (e) Pull out at 1 inch every 10 seconds.
   (f) Let the wafers cool briefly before proceeding to spin-on glass strip.

7. Check resistivity of control wafer.
   (a) Strip the spin-on glass from a single control wafer using the following instructions.
   (b) Measure the resistivity of this control wafer. It should have decreased about two orders of magnitude to $\approx 4\,\Omega/\square$.
   (c) An unexpectedly high resistivity (i.e., close to the pre-doping resistivity value) implies the doping may not have completed. To fix this, the device wafers (and the single remaining control wafer with spin-on glass—this is why there are two!) can be returned directly to the furnace, or stripped/have dopant respun/returned to the furnace again.

8. Strip spin-on glass.
   (a) After wafers are cooled, dip in 10:1 BHF.
      - Etch rate of spin-on glass is about $4700\,\text{Å}\,\text{min}^{-1}$. An etch time of about 2.5 min should suffice. Etching for too long will remove significant field oxide.
      - Ensure that you use 10:1 BHF; 5:1 is too fast and will significantly etch field oxide.
      - Wafer may not be hydrophobic due to film left by spin-on glass. Piranha will remove this film.
      - Rinse in DI water for 1 minute in each of 3 successive beakers.
      - Blow dry.

Step 7: Dopant Drive-in & Intermediate Oxidation

MEASUREMENTS: furnace temperature, oxidation time, anneal time, intermediate oxide thickness, resistivity of doped control wafer(s) after intermediate oxide strip.
CONTROLS: The control wafers are extremely important in this step. There should be at least one control wafer doped in the same way as the device wafers (two, if doping worked successfully the first try in the previous step). This wafer will be processed in the same way (have its dopant diffused), then have its resistivity measured to determine the state of the dopants.

1. Clean wafers (and control wafer(s)—if only one control wafer with the same doping as the device wafer remains, only use that one) with piranha following the the standard pre-furnace methods as in Step 3 on page 9.
    - We don't want an HF clean here: there's no need to remove native oxide (we're just growing more!), and we don't want to overetch the field oxide.
2. Drive-in and wet oxidize wafers (and control(s)) in furnace tube #2 at $1050\,^{\circ}$C.
    (a) Prepare the furnace bubbler, which creates water vapor for the furnace. This should be done by TA before the wafer clean.
        i. Fill bubbler with water using a clean, well rinsed beaker. The water level should be significantly above the bottom end of the tube in the bubbler apparatus which admits steam into the gas flow to the furnace.
            - **NOTE:** This water will evaporate over time. Check it periodically; it will probably need to be refilled once or twice during the lab.
        ii. Turn on heater tape. This heats the tube to the furnace so water vapor does not condense in it.
        iii. Turn on bubbler heater to 98 °C.
        iv. Make sure the release valve (cap) on the bubbler is loosely capped, or open the cap in case the steam pressure built up in the bubbler might push water into the furnace. Non-greased aluminum foil can be used as a makeshift bubbler cap if you are having issues.
    (b) Set gas flow for dry oxidation: bottom center knob at "furnace", top left knob to "bubbler", top center knob straight up, O_2 flowmeter at 65 sccm, and N_2 flowmeter to 0 sccm (off).
    (c) When the water is boiling in the bubbler, push wafers into furnace at the normal slow rate (1 inch every 10 seconds).
    (d) Set gas flow for wet oxidation. Set O_2 flowmeter at 25 sccm, and turn bottom center knob to "bubbler".
    (e) Allow wafers to wet oxidize for approximately 37 min (determined by TAs).
        - Wet oxidation (versus dry) is used because it grows oxide faster.
        - This EE143 furnace grows significantly less oxide than expected by Deal-Grove model, maybe due to low partial pressure of water vapor from the bubbler.

    - Previous versions of this EE143 manual used lower oxidation times (then allowed the wafers to remain in the furnace to anneal, for a total of $37\,\text{min}$).
    - Comment: used $25\,\text{min}$ in Fall 2013 and thickness varied widely $750\,\text{Å}$ to $9000\,\text{Å}$ run-to-run. Used $8\,\text{min}$ to $10\,\text{min}$ in Fall 2016. Unexpectedly slow in Fall 2022 and Spring 2023: used $30\,\text{min}$ for $900\,\text{Å}$.

   (f) Turn botton center knob from “bubbler” to “furnace”, turn on N_2 to $40\,\text{sccm}$, and turn off O_2 flow.

   (g) If wafers were in furnace for less than about $37\,\text{min}$, leave the wafers in the furnace (which should now only have nitrogen) to anneal until that time is reached. This time determines the amount dopants diffuse into the wafer.

   (h) Pull wafers out in N_2 at 1 inch/10 seconds.

   (i) Turn off bubbler and heater tape. Leave furnace at $N_2 = 120\,\text{sccm}$.

3. Measure oxide thickness on control wafer(s) with Nanospec.

4. Strip oxide from control wafer(s) and measure its sheet resistance with the four point probe. This is an indicator of the source/drain doping concentration (and resistivity).

Step 8: Contact Hole (CONT) Photolithography & Etch

MEASUREMENTS: etch time, pictures of CONT line/space structure (a) in photoresist and (b) after PR strip, pictures of CONT-ACTV and CONT-POLY alignment marks.
CONTROLS: Control wafers are not used during this step.

1. Apply photoresist to the wafer, expose it, and develop via the usual process (see Step 2 page 6).
   - Use the CONT mask, aligned primarily to the ACTV layer (CONT-ACTV alignment is slightly more sensitive than CONT-POLY alignment).
   - This alignment is a bit tricky because the CONT mask is dark field.
2. Save microscope images of the CONT line/space, CONT-ACTV alignment, and CONT-POLY alignment patterns for later analysis.
3. Etch away the oxide in contact holes with 5:1 BHF.
   - Use the same procedure as for the field oxide etch in Step 2 on page 7.
   - Etch for long enough to etch through the field oxide and/or gate oxide, as there are contacts on both. A ≈ 5 min etch is a good starting point.
   - **CAUTION:** Be sure to etch through all the oxide so that metal in the next step can contact the silicon underneath! An overetch (50% or even 100%) is strongly preferred to an underetch for this step, as it will merely result in larger contacts, not broken devices.
4. Inspect the wafer to ensure the etch completed.
   - Again, it is important the silicon is exposed so that metal makes good contact!
   - Although it is a bit tricky to tell when the etch is complete, contact holes on the silicon substrate (not the poly) appear white when cleared. Also, you may see the oxide etched out from underneath the edge of some of the photoresist.
   - If need be, you can try removing the resist and inspecting wafer again. If the contacts are not clear, etch for an additional 15 seconds and check again.
5. Strip photoresist with Remover 1165 using the same process as in Step 2 on page 8.
6. Save a microscope image of the CONT line/space pattern for later analysis.

Step 9: Metallization

MEASUREMENTS: aluminum sheet resistance.
CONTROLS: Preferably, aluminum is evaporated onto a control wafer so the aluminum resistivity can be measured. It does not matter much whether the wafer is oxidized or not because the aluminum conductivity is so much higher than that of the silicon.

1. Clean the wafers as for the furnace as in Step 3 on page 9 (piranha & HF). The goal is to remove any contaminants and native oxide so the aluminum directly contacts the silicon surface.
    - Clean the wafers just before they are put into evaporator in order to minimize the time native oxide can grow. Note the Al evaporator can only accommodate 3 wafers each time.
    - Don't overetch the field oxide!
2. Evaporate aluminum. Most steps are performed by TA.
    - (a) Check the pump: ensure the turbo pump "Normal Operation" light is on; otherwise, press the start button on the turbo pump controller and the light will be on within a few minutes. You can test the pump at this point by tuning the knob to PUMP. The chamber pressure is decreased through the roughing valve, then will automatically cross over to high vacuum. System will pump indefinitely in this mode.
    - (b) Vent the chamber: turn the knob to VENT. This closes all valves and vents N_2 into the chamber. After the chamber reaches atmospheric pressure, the jar will lift from the steel base. Lift the jar up.
    - (c) Load evaporator.
        - i. Lift the stainless steel wafer holder out of the inner glass cylinder ("chimney") and place it on a technicloth. Place wafers facing down on top of wafer holder. Note this evaporator can only hold 3 wafers at once (also, a wafer must be put into every slot to prevent metal coating the glass jar. Use dummy or control wafers if need be).
        - ii. Make sure to include something that can later be placed into the four point probe to measure the resistivity of the deposited aluminum film. This is preferably a control wafer, though you can also use kapton tape to attach a glass slide facing the aluminum.
        - iii. Place a clean glass slide in the chimney side for a clear window.
        - iv. Put about 6 Al source pellets on a tungsten boat into the evaporator.
            - Most of this aluminum will evaporate away; some will wick into the boat.
            - The tungsten boat can be reused several times but will eventually break.
        - v. Turn shutter knob so that the shutter is not covering the Al source.
            - Ideally the shutter would cover the source until it is fully heated to prevent contaminants (e.g., aluminum oxide from the pellet surface) from evaporating, but evaporation happens so fast there's not much point.

        vi. Replace wafer holder back inside the chimney.
        vii. Wipe stainless steel base and bottom of bell jar with technicloth soaked with IPA to ensure good vacuum seal.

    (d) Pump chamber to vacuum: lower bell jar and turn switch to PUMP. Push down bell jar cage until vacuum begins to hold it tight. The rough vacuum pump will remove most of the air, then the high vacuum turbopump will be automatically connected ("crossover") to the chamber to lower the pressure further.

        - After crossover, turn on the high vacuum gauge by pressing the "Gauge" switch. Note it is a toggle; the switch returns to its original position.
        - Ideally the vacuum would reach 2×10^{-6} torr to 5×10^{-6} torr. At this pressure, the flux of the residual gases (mostly H_2O) is about 1% of the Al flux during deposition. However, it takes the vacuum pump a long time to reach this pressure.
        - **NOTE:** To finish in a reasonable time, wait only until the pressure is below 1×10^{-5} torr. This is the highest pressure at which evaporation will physically work, because the mean free path is larger than the size of the evaporation chamber.

    (e) Turn off the ion high pressure gauge before starting the evaporation.

        - Al will initially react with water vapor physisorbed to the chamber walls ($2\,Al + 3\,H_2O \longrightarrow Al_2O_3 + 3\,H_2$), causing the ion gauge to jump several decades if left on, which can cause damage to the thin tungsten filament inside the gauge.

    (f) Switch evaporator (electrode) power to ON. Increase power by turning knib slowly clockwise. Units are in amps. At ≈ 40 A coil begins to glow, but the Al pellets do not melt. This can be observed through the slide window. Let heat at ≈ 40 A about 20 s to drive off water vapor.

    (g) Increase electrode power to ≈ 60 A (it may need to be a bit higher). The aluminum will evaporate quickly and the slide will start to be coated with Al.

        - If the tungsten boat is worn down or the power is too high, the current will heat the thinnest part of the boat so much that it vaporizes (the same way a fuse works!), breaking the circuit and stopping evaporation. If this happens, stop evaporation, replace the boat, and redo evaporation as necessary.

    (h) Slowly turn down power then switch electrode power to OFF.

    (i) Remove wafers in the same way they were loaded.

    (j) After wafers are removed, pump chamber to vacuum (following the same instructions as when pumping chamber before deposition). Leave evaporator in this state.

3. Use four point probe to measure sheet resistance of deposited aluminum on control wafer (or whatever other surface was used). This can be used to calculate the aluminum thickness.

    - The aluminum should be an opaque silver film.
    - We want the aluminum thickness to be greater than about 100 nm. Thinner aluminum is a bit transparent, fragile, difficult to probe, and has higher resistivity than in bulk.

Step 10: Metal (METL) Photolithography & Etch

MEASUREMENTS: etch time, pictures of METL line/space structure (a) in photoresist and (b) after PR strip, picture of METL-CONT alignment mark, furnace temperature & time.
CONTROLS: Control wafers are not used during this step.

1. Apply photoresist to the wafer, expose it, and develop via the usual process (see Step 2 page 6). Use the METL mask, aligned to the CONT layer.
2. Save microscope images of the METL line/space and CONT-METL alignment patterns for later analysis.
3. Hard bake the photoresist-coated wafer in a $110\,^\circ$C oven for 10 min.
4. Etch the aluminum (using PR as mask):
   (a) Prepare ≈ 1000 mL of Al etchant in a glass beaker. Heat in water bath on $115\,^\circ$C hot plate; allow 10 min for temperature to stabilize before use. The goal is to heat the Al etchant to $40\,^\circ$C to $50\,^\circ$C. Use stir bar to improve circulation and temperature uniformity. Note: hot plate in acid hood is on physical timer to the right of hood.
   (b) Immerse wafer in water for at least 10 seconds to wet surface.
   (c) Place wafer in aluminum etchant.
      - The etch rate of Al is $\approx 530\,\mathrm{nm/min}$ at $50\,^\circ$C. Be careful not to overetch!
      - Bubbles will form on the surface of the wafer as the etchant removes the metal. Agitate the wafers to ensure consistent supply of reactants to the wafer's surface. Bubbling will subside as the etch reaches completion.
      - Al etchant has a composition of approximately 80% Phosphoric acid, 10% H_2, 5% acetic acid, and 5% nitric acid.

   (d) Rinse wafer in 3 successive water baths.
   (e) Inspect wafer under microscope to ensure etch is complete.
5. Strip photoresist with Remover 1165 using the same process as in Step 2 on page 8.
6. Save a microscope image of the METL line/space pattern for later analysis.
7. Sinter wafers (done by TAs).
   (a) Check that the forming gas cylinder is not empty!
   (b) Do not clean the wafers with piranha, which attacks aluminum.
   (c) Place wafers in furnace tube 1 (top) at $400\,^\circ$C in forming gas (90% N_2, 10% H_2) for $20\,\mathrm{min}$. Gas flow set to 40 sccm. Slow push/pull is not necessary.
      - Sintering does two things. It allows the aluminum and silicon to interdiffuse, forming a good contact. It also allows hydrogen atoms from the forming gas to diffuse into the silicon and tie up surface states at the oxide channel interface.

Congratulations!!!

The fabrication process is now completed,
and the devices are ready for testing!

See the EE143 Characterization Manual for details.

Appendix: Cleaning

Mask Cleaning Procedure

At sinks douse chrome side of mask with acetone or mask cleaning solution. If necessary one may lightly use a mask scrubber to remove any residual resist. Exercise extreme caution if you choose to use the scrubber so that the mask is not damaged. While acetone is still pooled on mask, use IPA (2-propanol) to rinse acetone off. Do not use water! Blow mask dry using N2 gun.

Furnace TCA Clean

Before the lab session, NanoLab staff should TCA clean the furnace.

1. Start with the furnace already on and stabilized. Confirm temperature controller is set to the required temperature. For $1100\,^{\circ}$C the controllers should be about +30.0, 87.0, +00.0. The center value adjusts the temperature of the central zone of the furnace. The left and right values set the temperature of the load and gas-input zones relative to the center region. Check the temperature in the "hot zone" using the thermocouple.

2. Open the sliding exhaust (scavenger) door on the side of the loading vestibule. Close the others if they are not being used.

3. Remove the glass end cap from tube (holding it with an insulating glove) and place it on the fire brick on the counter. Load an empty boat into the "hot zone". Push boat in no faster than 1" every 10 seconds. Pushing the boat in faster can cause the boat to crack due to thermal stress. Replace the glass end cap. Make sure gas outlet is pointing towards the exhaust (scavenger) door.

4. Turn on O_2 flow at maximum rate (15+ cm, steel ball) for at least $10\,$min. This requires that the O_2 tank is turned on, the regulator is set to 10 psi (MAX), the postregulator valve is on, the valve on the flow meter is opened to read 15 cm+, and the O_2 valve at tube #3 is opened. *****This step is critical; if there is not sufficient oxygen in the tube when the TCA starts flowing, the TCA will not oxidize (burn) completely and will form soot inside the tube.*****

5. Start the TCA bubbling by closing the N_2 valve at tube #3, opening the TCA valve at tube #3, opening the valve to the TCA bubbler, and opening the N2/TCA valve on the flow controller. Keep this to a low flow rate, say 1-2 on the scale. It is important to open valves starting at the tube end. Flowing gas into the bubbler vessel with the tube 3 TCA valve off will pressurize the bubbler vessel, causing it to explode. This would be very bad. Leaving the tube #3 N_2 valve on will cause N_2 gas to circumvent the bubbler vessel. If bright blue flames are visible at the tube inlet, decrease the N_2 flow rate. The flames are a result of too much free carbon in the tube, i.e. not enough O_2 to react with the incoming TCA. Too much TCA will cause a small explosion. (This has happened before. The other end of the tube was blown off.) If no bubbles are seen, check that the TCA level is above the frosted zone of the N_2 tube in the bubbler vessel.

6. TCA clean for 1 hour at $1100\,^{\circ}$C. TCA removes heavy-metal contaminants by supplying the chlorine to form volatile chlorides: $N_2 + 2\,O_2 + C_2H_3Cl_3 \longrightarrow 2\,CO_2 + 3\,HCl + N_2$.

7. Turn off N_2/TCA flow, and close TCA bubbler valve, and the tube #3 TCA valve. It is important to close valves starting at the flow meter, and working towards tube #3, thereby not pressurizing the TCA bubbler.

8. Wait 10 minutes after turning off TCA.

9. Switch gases to $N_2 = 40\,$sccm and $O_2 = 15\,$sccm for wafer loading. This is 90% N_2 and 10% O_2.